

Serial No.: 10/036,877  
Amdt. Dated: September 30, 2004  
Reply to Office action of July 27, 2004

RD-29276-1

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**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Application of: James Manio Silva

Group Art Unit: 1724

Application No.: 10/036,877

Examiner: Cintins, Ivars C

Filed: January 4<sup>th</sup> 2002

Confirmation No.: 5166

For: METHOD FOR PURIFYING BRINE

**DECLARATION OF APPLICANT UNDER 37 CFR §1.132**

Mail Stop AF  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

S I R:

In support of the Amendment submitted concurrently herewith, I the undersigned,  
declare as follows:

- 1) That I am James Manio Silva an inventor of the subject matter claimed in  
the above-identified U.S. Patent Application;
- 2) That I evaluated three methods of brine purification, Methods 1-3, below  
wherein the brine was subjected to passage through a series of resin treatment columns in  
the order A, B, C shown to provide three purified brine samples:

Serial No.: 10/036,877  
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RD-29276-1

**Method 1:**

- A. AMBERSORB 572 (pH 3.5, 2 bed volumes/hr)
- B. Transition Metal Cation Removal (pH 3.5, 2 bed volumes/hr)
- C. Hardness Removal (pH 10.4, 2 bed volumes/hr)

**Method 2 :**

- A. AMBERSORB 572 (pH 3.5, 2 bed volumes/hr)
- B. Transition Metal Cation Removal (pH 3.5, 2 bed volumes/hr)
- C. Hardness Removal (pH 10.4, 2 bed volumes/hr)
- D. AMBERSORB 572 (pH 10.4, 2 bed volumes/hr)

**Method 3:**

- A. Transition Metal Cation Removal (pH 3.5, 2 bed volumes/hr)
- B. Hardness Removal (pH 10.4, 2 bed volumes/hr)
- C. AMBERSORB 572 (pH 3.5, 2 bed volumes/hr)

3) That each of the purified brine samples purified by Method 1-3 was subjected to essentially the same the electrolysis conditions for a total of at least 23 hours over which times the voltage penalties observed were in 3.6 mV/hr, 1.2 mV/hr and 0.05 mV/hr respectively;

4) That Method 1, the Comparative Example, showed a high and unacceptable voltage penalty of 3.6 mV/hr after only 23 hours of operation whereas Methods 2 and 3, which are embodiments of the instant invention, showed excellent performance on the basis of the relatively low voltage penalties observed when Methods 2 and 3 were employed;

5) That the data provided in Table 1 of the attachment submitted herewith, offers proof of the unexpected superiority of Methods 2 and 3;

6) That the unacceptably high voltage penalty observed when Method 1 was employed was itself unexpected in view of the references, which are silent on the precise order of the treatment steps, demonstrated herein;

Serial No.: 10/036,877  
Amdt. Dated: September 30, 2004  
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RD-29276-1

7) That all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and

8) That these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon;

9) Further Declarant sayeth not.

Respectfully Submitted,

By James Manio Silva  
James Manio Silva

Dated Sept. 30, 2004

Attachment: I

Serial No.: 10/036,877

Amdt. Dated: September 30, 2004

Reply to Office action of July 27, 2004

RD-29276-1

## ATTACHMENT I

\*TABLE 1

Example	Brine	ppm Sodium Gluconate	Treatment	Voltage increase Rate
1	Ultrapure	0	Method 1	3.6 mV/hr
1	Ultrapure	0	Method 2	1.2 mV/hr
1	Recycle	0	Method 3	0.05 mV/day

## EXPERIMENTAL DESCRIPTION OF HOW THE TESTS WERE CARRIED OUT:

## Example 1-Method 1 [Comparative Example] paragraph 72 lines 1-9

Gluconate-free, "clean" brine was prepared from Morton CULINOX 999 food grade salt and dionized water. Brine which is free of gluconate ions and is made from food grade salt and deionized water is referred to as "clean" brine. This brine was subjected to brine purification Method 1 using fresh ion exchange resins to yield a purified brine. The purified brine was then fed to a laboratory membrane electrolyzer that had been previously conditioned by operation with ultrapure brine. The cell voltage was 3.400 volts just prior to switching from ultrapure brine to the purified brine. After 23 hours of operation with the purified brine, the voltage had increased by 83 mV (3.6 mV/hr).

## Example 1-Method 2 [Example] paragraph 72 lines 9-15

The laboratory membrane electrolyzer cell was then switched to a feed of the purified brine [of Example 1-Method 1] which had been further subjected to treatment by passage through a bed of AMBERSORB 572 resin at pH 10.4 and a rate of 2 bed volumes/hr to give a "polished" brine. The purification and polishing steps used here constitute brine purification Method 2 which is said to afford a "polished" brine. The cell voltage for the brine treated using Method 2 increased from 3.400 volts to 3.428 volts after 24 hours (1.2 mV/hr).

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RD-29276-1

**Example 1-Method 3 [Example]**

Recycle brine containing 0 ppm gluconate was purified by the "primary brine treatment" (See Application paragraph 36) and then subjected to Method 3 and fed to a lab membrane cell that had been conditioned by operation with ultrapure brine. The recycle brine following the primary brine treatment had the same brine strength (i.e. concentration of sodium chloride) as the ultrapure brine but contained the following impurities which are listed below.

Impurity	Recycle Brine after Primary Brine Treatment
Calcium ion	0.6 ppm
Magnesium ion	0.2 ppm
Iron ion	0.3 ppm
Silica	10.8 ppm
Na <sub>2</sub> SO <sub>4</sub>	160 ppm

In addition, recycle brine contained about 6.5 ppm of the quaternary ammonium salt, chloromethyltriethyl ammonium chloride. The ultrapure brine contained no quaternary ammonium salt. The cell voltage was 3.330 volts just prior to switching from ultrapure brine to the recycle brine which had been subjected to primary brine treatment and the brine treatment of Method 3. After 30 days of operation with purified brine, the voltage was 3.366 volts (0.05 mV/hr). This example shows the advantage of Method 3 over Method 1 for brines that do not contain gluconate.